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Profile coating for KB mirror applications at the Advanced Photon Source

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ABSTRACT

For microfocusing x-ray mirrors, an ellipse shape is desirable for aberration-free optics. However, it is difficult to polish elliptical mirrors to x-ray quality smoothness. A differential coating method to convert a cylindrical mirror to an elliptical one has been previously reported. The differential coating was obtained by varying the sputter source power while the mirror was passed through. Here we report a new method of profile coating to achieve the same goal more effectively. In the profile coating, the sputter source power is kept constant, while the substrate is passed over a contoured mask at a constant speed. The mask is placed very close to the substrate level (within 1.0 mm) on a shield-can over the sputter gun. Four-inch-diameter Si wafers were coated through a 100-mm-long by 152-mm-wide aperture on the top of the shield-can. The thickness distribution was then obtained using a spectroscopic ellipsometer with computer-controlled X-Y translation stages. A model has been developed to fit the measured thickness distribution of stationary growth. The relative thickness weightings are then digitized at every point 1 mm apart for the entire open area of the aperture. When the substrate is moving across the shield-can during a deposition, the film thickness is directly proportional to the length of the opening on the can along the moving direction. By equating the summation of relative weighting to the required relative thickness at the same position, the length of the opening at that position can be determined. By repeating the same process for the whole length of the required profile, a contour can be obtained for a desired thickness profile. The contoured mask is then placed on the opening of the shield-can. The number of passes and the moving speed of the substrate are determined according to the required thickness and the growth-rate calibration. The mirror coating profile is determined from the ideal surface figure of a focus ellipse and that obtained from a long trace profiler on the mirror. Preliminary test results using Au as a coating material will be presented.

Keywords: x-ray optics, KB mirrors, microfocusing, sputter deposition, profile coating, LTP metrology, ellipsometry

1 INTRODUCTION

Modern science using synchrotron radiation x-rays requires smaller and smaller beams to achieve unprecedented spacial resolution. A great deal of effort has been put in place to improve x-ray microfocusing, especially in applications of Fresnel zone plates and Kirkpatrick-Baez (KB) mirrors. The KB mirrors have the advantage over zone plates of a longer focusing length and a broader bandpass. The KB mirrors are two concave mirrors at glancing angles to the x-ray beam and arranged 90° to each other to successively focus x-rays in both the vertical and horizontal directions. In reflective optics, elliptical mirrors are desirable for microfocusing. It is well known that a ray in any direction from one focal point in an ellipsoid will be reflected into the other focal point.^[1,2] Cylindrical or spherical mirrors unavoidably will cause aberration. Sophisticated bending techniques have been developed to bend Rh- or Pt-coated thin Si plates to achieve a desirable elliptical shape for microfocusing. Submicron x-ray beams have been achieved by using benders for KB mirrors.^[3,4] We still have sizable orders of Rh or Pt coatings at our deposition lab for bendable KB mirrors.

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The KB mirrors with benders are flexible in adjusting the focal length, but the benders are bulky and the mirrors are difficult to cool. Monolithic KB mirrors would be much easier to use if one could obtain the desired elliptical surface profile. As a result, people are looking for ways to achieve monolithic elliptical mirrors. One way is to directly polish the Si mirror surface to an elliptical profile, which is generally very hard to do by using the standard chemical-mechanical polishing techniques because of the demanding requirements of small figure errors and root-mean-square (rms) roughness. For x-ray microfocusing, submicroradian figure perfection from an ideal ellipse and a $< 3 \text{ \AA}$ rms surface roughness are required. Most recently a new micromachining technique that combines plasma chemical vaporization machining, elastic emission machining, together with long-trace profiler (LTP) measurements has produced very encouraging results.^[5] Another way is by differentially depositing films on selected areas of a well-polished spherical or cylindrical mirror as previously reported.^[6] The technique for polishing such symmetric mirrors is mature, and mirrors can be polished more economically in large quantity. Differential deposition can then be applied to modify the surface figure to a desired profile. The previously reported differential deposition technique uses a narrow slit in front of the mirror while varying the power of the sputter gun as the mirror is passing across the slit. The power of the sputter gun for each mirror position is determined according to the thickness requirement calculated from LTP measurements for that position. To be effective, the programmable ramp of the power supply is limited to its linear range. The slit width cannot be too small because of the diffraction effect between the sputtered atoms. To increase the controllability of the process, the target is kept $\sim 8''$ away from the mirror surface.^[7] This method requires a fine control of the overlap of the neighboring Au coatings. Both the micromachining and the previously reported differential deposition techniques need many cycles of measuring and machining/deposition tries.

In this paper we report a new profile-coating technique to achieve the same goal of making good elliptical KB mirrors more efficiently. It would be wonderful if one could convert an ordinary cylindrical mirror into a x-ray quality elliptical mirror by one Au deposition. Gold-coated mirrors have the added advantage of increased critical angle of total external reflection as compared to bare Si mirrors. This paper shows that it is possible to make such elliptical mirrors by using the profile-coating technique.

2 PROFILE-COATING TECHNIQUES

The profile-coating technique evolved naturally at the Advanced Photon Source (APS) deposition lab. The deposition facility consists of four large vacuum chambers, each 16 inches in diameter and 66 inches long. Three CTI model CT-8 cryo pumps and an Alcatel ADP 81 dry pump provide a base pressure of $< 1 \times 10^{-8}$ Torr for the system. Samples on a sample holder can be loaded into a carrier, which can be moved from chamber to chamber by a computer-controlled transport system. Four 3-inch-diameter magnetron sputter guns are deployed in the deposition chamber. The sputter targets are facing up, and the substrates are facing down. During the deposition, the substrates are usually moving. We have used masks and the linear substrate motion to improve the uniformity of coatings. The mask is placed close to the substrate level on a shield-can over the sputter gun. Uniform deposition can be achieved through the design of a shaped-aperture mask over the sputter gun. Later we used this technique to make laterally graded W/C multilayers, where the W layer was kept uniform in thickness and the C layer had a wedge shape.^[8] In both cases of uniform and graded coatings, the profile of the film thickness is known and well defined. The same technique is improved to make profile coatings for elliptical mirrors. In this application, a desired surface profile after the profile coating on a cylindrical mirror should be the ideal surface figure of a focus ellipse. The coating profile is then determined by the difference between the ellipse profile and that measured profile of the cylindrical mirror from a long trace profiler. Every mirror requires a different coating profile. Also, the profile is usually not mathematically well defined, so that it can only be determined through point-by-point calculations. According to our experience, a profile calculated for each position 1 mm apart is sufficient. A spline fit of the calculated data points then provides a smooth curve. In the following we present the basics of the profile-coating technique.

2.1 How to make the desired mask

First of all, one needs to know how the sputtered atoms are distributed on the area above the sputter gun at the substrate level. Film-thickness distribution in magnetron sputtering has been extensively studied in the past few decades.^[9, 10] Generally the thickness t of the film deposited from a ring source onto a flat stationary substrate can be expressed as

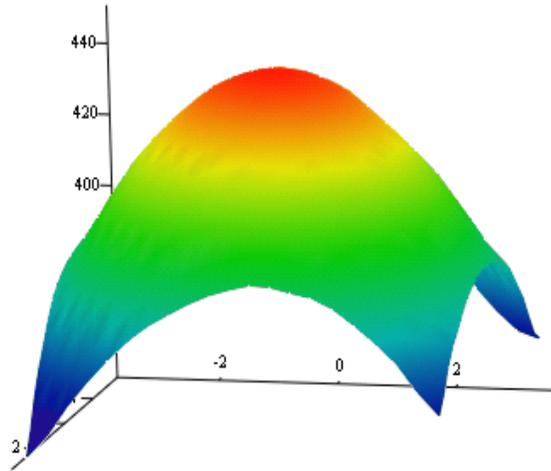
$$t = m_x h^2 (h^2 + r^2 + a^2) / [\rho \pi (h^2 + r^2 + a^2 + 2ar)^{1.5} (h^2 + r^2 + a^2 - 2ar)^{1.5}], \quad (1)$$

where m_x is the mass of emitted material on the ring source, ρ is the density of the material, h the source-to-substrate distance, r the radius of the source ring, and a the position on the substrate.^[10] The relative thickness distribution is simply t/t_0 , where t_0 is the thickness at $a=0$. Here h and r are known from the experimental setup and the erosion profile of the target. In the case of Au coating for KB mirrors, we had $h = 11.4$ cm and $r = 2.3$ cm. The ratio of t/t_0 is thus a fixed function of a . In practice, however, there are usually some deviations from measured thickness profiles. In order to fit the experimental data, an extra term of $f(a)$ is added. This term is obtained from a polynomial fit of the difference between the experimental curve and that of t/t_0 from Eqn. (1). The final model for the relative thickness distribution is $[1+f(a)] * t/t_0$.

The experimental data for thickness distribution were obtained as follows. First, any existing mask from the shield-can was removed. Then a thin film (~40 nm thick or less) was grown on a stationary 4"-diameter Si wafer at a level where the KB mirror would be coated through the 100 x 152 mm² aperture on the top of the shield-can. The film thickness distribution was obtained by using a M-44 spectroscopic ellipsometer equipped with an automated X-Y translation station.^[11] Figure 1 shows such a thickness distribution for an Au film. It was obtained by measuring the thickness every 1 mm apart along both the x and y directions.

Using the above model, one can then obtain the deposition weighting for any position above the 100 x 152 mm² aperture on the top of the shield-can at the substrate level for Au profile coatings. The length of the aperture is 100 mm along the moving direction, and the width is 152 mm. When the substrate is moving across the shield-can, the film thickness is directly proportional to the length of the opening of the can along the moving direction (with a maximum of 100 mm). By equating the summation of relative weighting to the required relative thickness at the same position, the length of the opening at that position can be determined. By repeating the same process for the whole length of the required profile, a contour can be obtained for a desired thickness profile. For example, the thickness profile for a uniform coating is a straight horizontal line in a thickness vs. position plot. The mask profile is then determined by choosing a length of 100 mm (or less) at positions of ± 76 mm, calculating the total weighting at this position, and calculating the length needed at other positions in order to have the same total weighting as that at the 76-mm position. To simplify matters, symmetry is used in the calculations and should be ensured in the experimental setup. Figure 2 shows a mask for uniform Au coating on top of a shield-can over the Au target. Figure 3 shows the result of a test run measured using the ellipsometer. Good uniformity within $\pm 0.15\%$ is achieved.

For masks that are used to achieve a desired Au thickness profile on a cylindrical mirror for ellipse focusing, the coating profiles are obtained from LTP measurements. The mask profile can be calculated using the same procedure as outlined above, as long as the coating profile is known. The masks are cut from aluminum plates.



(x,y,t)

Fig. 1. Thickness distribution obtained from ellipsometer measurements of a gold film on a Si wafer placed directly above the Au target. The units are "angstrom" for the vertical axis and "cm" for the horizontal axes.

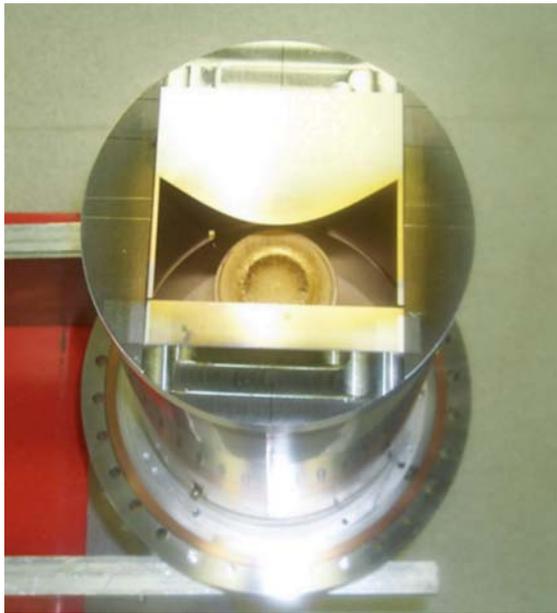


Fig. 2. A mask placed on top of the shield-can above the Au target to achieve a uniform coating.

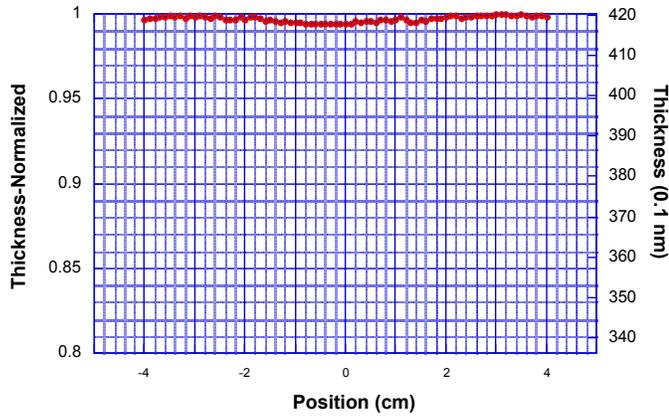


Fig. 3. Measured thickness profile for an Au thin film coated with the mask shown in Fig. 2.

2.2 How to obtain a coating profile from LTP measurements

The coating profile is the film-thickness distribution along the mirror's long direction. However, an LTP measures only the slope information of the mirror. As a result, the slope data need to be converted into height data. By simple one-sided rectangular integration one can convert slope data into a height profile. But as pointed out by Takacs et al.,^[12] this process may lead to artifacts if not carefully done. In this work the integration was performed using a trapezoidal rule, but fitting the measurements to the optimal ellipse was done by minimizing slope errors, without integration.

The LTP measurements were carried out at the APS metrology lab. The mirror substrate is 50 mm high, 20 mm wide and 90 mm long. It has a spherical surface profile with a radius of ~ 87 m along the long direction. The LTP sampling period on the surface was 1 mm, with the scan length usually set to start and end 2 mm from the ends of the mirror. To minimize random and systematic errors, three different sets of measurement data taken along the substrate main axis at three different locations were averaged. Each set of measurements was a result of averaging ten successive scans.

From the difference between the measured slope and that of a perfect ellipse one can obtain the coating profile. The input parameters of the ellipse correspond to the UNI-CAT 43-ID beamline at the APS. These parameters are: 36.5 m for the source-to-mirror distance, 2.6 mrad for the mirror glancing angle, and 130 mm for the mirror-to-focus distance.

Since the mirror angle is adjustable, one may choose a coating profile for minimum gradient at the center of the mirror or for minimum coating thickness.^[6] For the previously reported differential deposition, better performance was achieved using the profile where the deposition gradient is minimized at the center of the mirror. For better efficiency, we choose the coating profile where the deposition thickness is minimized. Figure 4 shows these two coating profiles together with a mask profile calculated from the coating profile that minimizes the coating thickness.

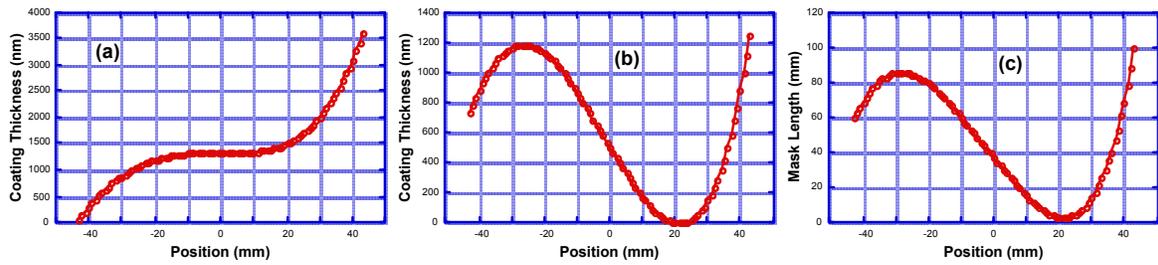


Fig. 4. Required coating thickness profile (a) with minimum deposition gradient and (b) with minimum coating thickness to achieve an ideal ellipse surface profile according to long trace profiler measurements, and (c) a mask profile calculated according to the coating profile (b).

2.3 Gold profile coatings

Gold is suitable for profile coating to convert a cylindrical mirror into an elliptical one. It was found that although Au initially grows as small islands on a Si substrate, thick Au films are usually smooth, especially when a thin Cr underlayer is coated first on the Si mirror.^[13, 14] In our experiments a thin Cr film of ~5 nm thick was used as a "glue" layer for better adhesion of the subsequent Au coating on Si. For profile coatings, it is also extremely important to load the mirror at the right position on the substrate holder. A test coating of a maximum Au thickness of ~40 nm on a ~12.5 mm x 100 mm Si strip was performed prior to the mirror coating. Then the film thickness was measured using the ellipsometer and normalized to compare with the required coating profile. Figure 5 shows the result of such a test run.

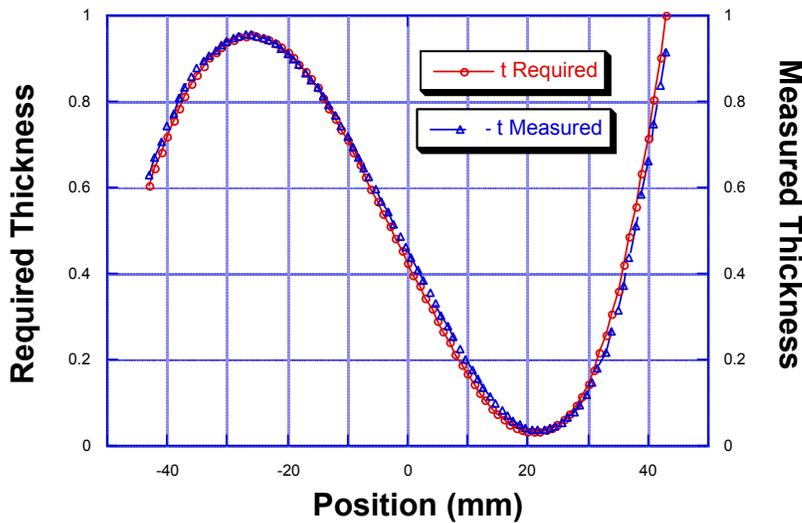


Fig. 5. Normalized required and measured thickness profiles for a test run of profile coating.

The test run serves two purposes. One is to check the profile with the required coating profile and pinpoint the maximum and minimum positions for mirror loading. The other is to obtain the scaling parameters for the final mirror coating. To obtain an elliptical profile, it is important to put down the right amount of Au at the right positions. Since ellipsometry can measure only a limited thickness range, the measurement can be done only on thin test samples. Fortunately, the magnetron sputtering process can be controlled to be very stable, and a linear scaling of the sample passing speed and number of passes is sufficient to achieve the right total thickness from the test results. This point of view has been confirmed by measuring a scaled-up, thick Au/Si sample using a TOPO interferometer.^[15] A thickness of 715 ± 9 nm was obtained from TOPO measurements. This result is in good agreement with the scaled-up number of 714 ± 8 nm from the result of an ellipsometer measurement on a thin Au/Si sample.

When the test was done, the mirror was carefully mounted on a mirror holder and loaded on the carrier so that the mirror surface is ~ 0.5 mm above the mask during the deposition. The whole deposition took less than an hour to complete. The coated mirror was then evaluated using the LTP measurements.

3 RESULTS AND DISCUSSIONS

Figure 6 demonstrates that an ordinary cylindrical mirror can be converted into an elliptical one by a single profile coating. It is the LTP result obtained after the coating as compared to an ideal elliptical surface. It shows that the desired elliptical shape has been achieved with an overall rms slope error of $1.656 \mu\text{rad}$ from an ideal ellipse. The surface figure is even better than that of the original cylindrical mirror, which has a $2.6 \mu\text{rad}$ rms slope error from an ideal cylinder. This result means that, by using the profile-coating technique, we can not only convert the slope of a mirror but also improve its figure error. From Fig. 6 one can see that, if we ignore the large slope error at the right end, a much better figure can be obtained, as shown in Fig. 7. In real applications, one has this freedom by moving the mirror to an optimum position.

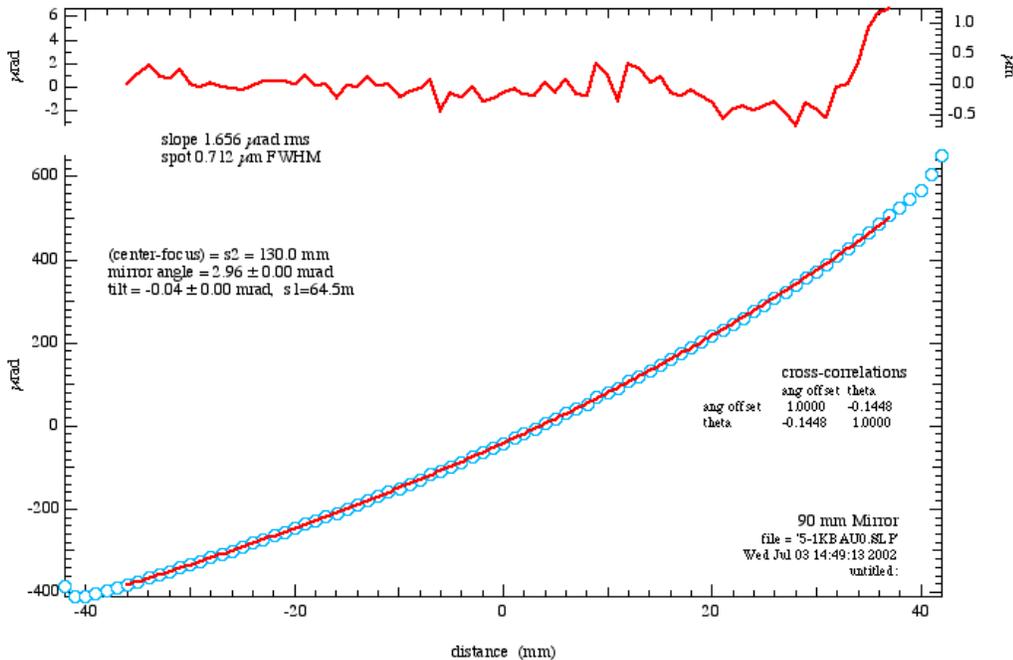


Fig. 6. A typical LTP measurement result showing 1) at the bottom, the measured slope as compared with an ideal slope and 2) on the top, the residual slope error as well as the rms number and the projected spot size of a focused beam. This figure shows the result for a mirror after one profile coating. The solid line on the bottom half is the slope of an ideal ellipse. The top shows the difference between the measured slope and the ideal slope, or the residual slope error. The large slope error on the right side is due to the edge effect of the mask.

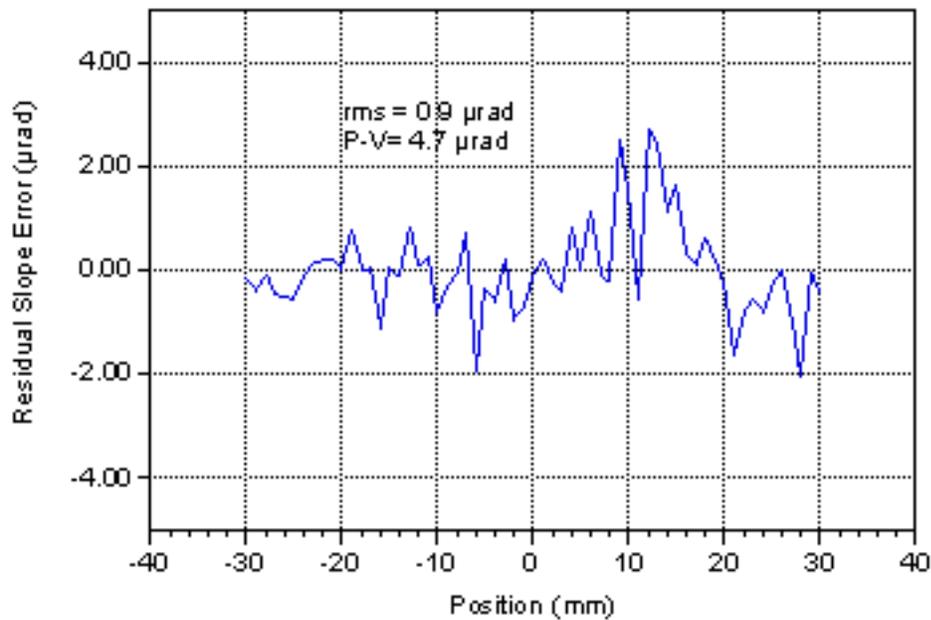


Fig. 7. The residual slope error of a portion of 60 mm of the 90 mm mirror from a perfect ellipse showing a rms value of $0.9 \mu\text{rad}$ after one profile coating.

These results demonstrate that the profile-coating technique is very promising for producing better KB mirrors. The technique is quite straightforward and effective in converting and improving the surface figure of ordinary cylindrical mirrors. These symmetrically polished mirrors can be made in quantity economically. The metrology testing of each x-ray mirror has to be performed regardless of whether the profile coating is used or not. The deposition process is fast and the target usage can be optimized. Also, for the Au-covered mirrors the critical angle of total external reflection is larger than that for the bare Si mirrors. Gold-covered mirrors can be made shorter because of the increased angle of incidence in reflective x-ray optics. Because of all these advantages, it is worthwhile to further improve this profile-coating technique.

This technique can be further improved on both the deposition and metrology fronts. In regard to deposition, it is important to improve the accuracy of system alignment and to venture different ways to reduce the edge effect caused by the use of masks. Fixing the sputter gun in position without wobbling was found to be important. The Au target and the shield-can need also be as concentric as possible. The positioning of the mirror in the mirror holder and the mirror holder in the transport carrier needs to be accurate with good repeatability. The positioning of the test sample and the mirror should also be as accurate as possible. The distance between the sample and the top of the mask should be as short as possible. But it is hard to go closer than 0.5 mm without running into the risk of scratching the mirror surface. This gap will introduce some shadowing effect. To reduce this effect, one needs to consider ways to compensate it in the mask fabricating process. We have continued to explore these fields.

As to the metrology front, we need to improve the accuracy of the measurement as well as the resolution. Without accurate height information about the mirror surface, it would be impossible to "fine tune" the profile-coating process to further improve the mirror quality. On the other hand, if we have this capability, we can further improve the quality of all KB mirrors, including bendable ones, by using the profile-coating technique.

4 SUMMARY

We have demonstrated that the new profile-coating technique can convert an ordinary cylindrical mirror into a x-ray quality elliptical mirror. Details of the profile-coating technique have been outlined in this paper. The excellent results and the possibilities for further improvements shown in this paper demonstrate that this technique is very promising for exploring the limits of achievable focus in x-ray optics.

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